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NEW METHOD OF INVESTIGATING THE CHEMICAL COMPOSITION OF A MICROVOLUME OF AN ALLOY

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ABSTRACT

The authors describe their equipment and method of analyzing the chemical composition of a microvolume of an alloy section from the characteristic X-ray radiation.

The development of solid state theory and also the practical problems /655* in producing alloys with specific properties require the development of special methods of investigating and analyzing the atomic electron structures and microcompositions of metals, alloys and compounds. Existing physical methods of investigation were directed in most cases to the investigation of atomic electron structures of solid bodies.

In 1951 one of the authors (ref. 1) in the USSR and simultaneously another author (ref. 2) in France proposed a new method of X-ray spectral quantitative analysis of composition at a "point." This method makes it possible to determine quantitatively most of the elements of the periodic table in a region of the order of several microns.

Subsequently we developed equipment and a method of investigation to analyze the chemical composition of a microvolume of an alloy section from the characteristic X-ray radiation excited by a focused electron beam in a volume of approximately 10 μ^3 with a sensitivity to 0.1 percent. This method determines the content of various elements at a given point of the section, as well as the distribution of a given element over different regions of the section.

*Numbers given in the margin indicate the pagination in the original foreign text.

In analyzing the composition at a "point" one of the approaches of conventional X-ray spectroscopy is utilized (ref. 3). The setup developed by us is shown in figure 1. An electron beam (EG, electron gun) is focused by one or two electromagnetic lenses L on the anode section of an X-ray tube A mounted in a special sample holder. The anode is displaced in the horizontal plane by means of special microscrews; one of the rotating levers contains a motor with a speed reducer to displace the anode section in a selected direction continuously under the electron beam with a velocity of 10-120 μ/min.

The holder is designed to hold simultaneously three samples, a flourescent screen and a frame with a grid for the control measurement of the focus distance. The X-ray tube contains the following: mirror 3 and a metallographic microscope M of original construction with a long focal length lens; the microscope itself is installed outside the vacuum. Its rigid mounting provides for the coincidence of the ocular cross hairs with the image of the focal spot on the anode section in the mirror. The electron current passing through the tube is measured by means of a Faraday cylinder connected to a dc amplifier.

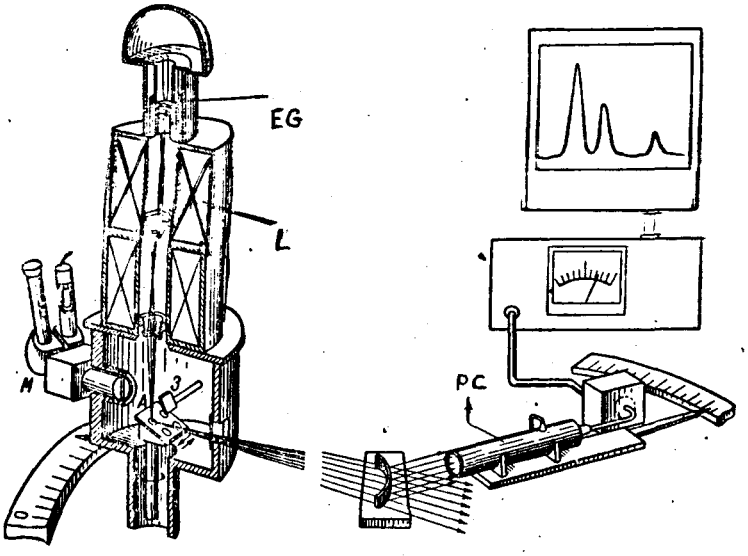

Figure 1

The X-ray radiation is broken down into the spectrum by means of a spectrograph with a bent crystal, using the rotation method for transmission. The stationary crystal is bent along a cylindrical surface with a radius of 300 mm. Two movable arms of the spectrograph, which are connected to each other, carry a microfocal tube and a photon counter (PC). The kinematic and vacuum device /656 displace the arm simultaneously by equal angles toward each other. Rotation is carried out manually or by means of a motor with a speed reducer and a speed reverser.

All high and low voltage sources are stabilized and the percent of stabilization is not lower than 0.05 percent. The high voltage supplied to the tube can vary from 30-50 kV. The electron current through the tube reaches a value of 1 μA. The diameter of the focal spot on the anode is 2-4 μ (when determined by the method of shadow photography of the screen made of wire with a diameter of 5 μ). The specific load on the anode section is equal to 1 kV/mm^2 on the average.

To record the intensity of the characteristic lines, a unit of the URS-50-I equipment is used to determine the intensity by computation or by direct reading and to record the spectrum by means of an electronic recording potentiometer. The different displacement velocities of the sample and of the chart paper /657 can vary the "amplification" of the recording over a wide range. The maximum "amplification" is 2×10^4.

During the optimum operation the intensity of the $K\alpha_1$ line of the pure element is 10^4-10^5 pulses/sec. Therefore, when the element to be determined is contained in large quantities, the operation at a given point of the section must take place with lower specific loads (smaller currents through the tube). The accuracy of the analysis which is determined by the stability of the voltage sources and by the recording circuit was established experimentally to be 2-5 percent.

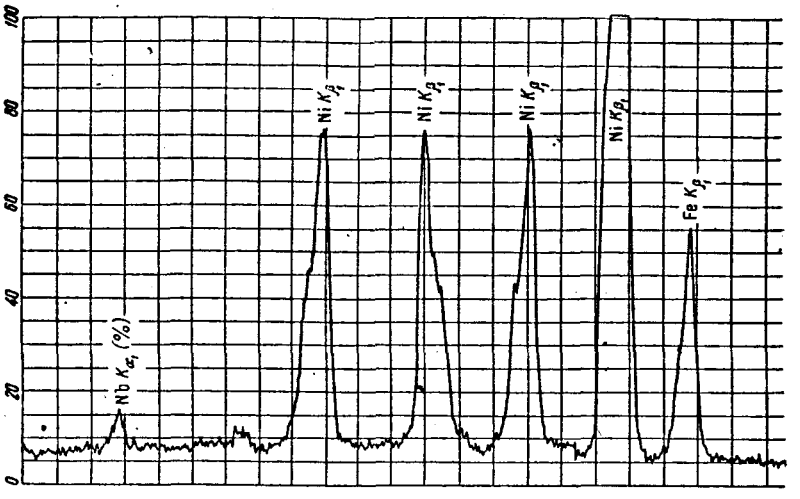

Figure 2

Figure 2 shows the spectrogram "at a point" of a multiple component alloy, illustrating the operation of our equipment.

Figure 3 shows the results of the "point" analysis of another multiple component alloy. Curve A shows the variation in the intensity of the Ni $K\alpha_1$ line (RC = 4 sec, scale 1,000 pulse/sec), when the section is displaced under the electron beam. Curve B shows the variation in the intensity of the line W $L\alpha_1$ (RC = 2 sec, scale 200 pulse/sec) at the same region of the section. A comparison of these data with the microphotographs of the section show that the observed inclusions on the section represent a phase which is substantially enriched with W and has a reduced content of the Ni base.

Thus the equipment and the method which we have developed can carry out a "point" quantitative analysis of the composition with a sensitivity to 0.1 percent (which corresponds to 10^{-13} g of the element "at the point") and can investigate the distribution of a given element along a section. A series of investigations was carried out on complexly alloyed metallic and cermet alloys, welding seams and diffusion layers for the distribution of the elements Fe, Ni,

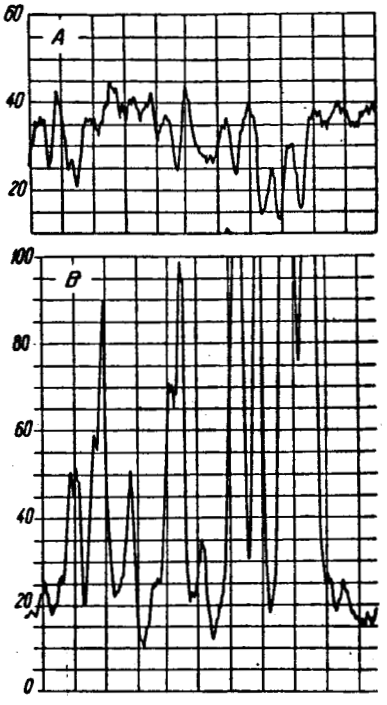

Figure 3

Cu, Zn, Nb, Mo, W, Re in microvolumes and in assigned directions. These investigations show that the new method has great practical possibilities in solving a series of problems which cannot be solved by any other existing methods of physical and chemical analysis.

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